Four additional hybrid poplars with papermaking properties superior to aspen

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ABSTRACT: Four hybrid poplars were compared to aspen and balsam poplar to determine their suitability for use in kraft and bleached refiner mechanical pulps (BRMPs). The four hybrid poplar kraft pulps had tensile properties superior to those of pulps from balsam poplar and aspen, the poplars traditionally used in papermaking. High densities for sheets prepared from the hybrid poplars indicated that the cell walls of their fibers were not as thick as those of the two traditional poplars. High strain and tensile energy absorption were found in the sheets derived from the hybrids. Three of the four hybrid poplar BRMPs had bleached brightnesses and light scattering coefficients equivalent to those of aspen. All four hybrid pulps had strength values almost equal to that of aspen pulp, even though their percentages of long fibers were much lower. The hybrid poplar chips were more rigid than those from aspen or balsam poplar, partly because of their higher lignin content.

Application: Equal in strength and bleachability to pulps derived from aspen, pulps from hybrid poplars have great potential in papermaking as traditional supplies of hardwood fiber diminish.

According to some projections, regional shortages of hardwood papermaking fibers may occur around the year 2010 [1,2]. The prospect of these shortages motivated several American paper companies to experiment with hybrid poplar plantations in the early- to mid-1990s. Examples are plantations owned by Potlatch Corp. [3], Boise Cascade Corp. [3], and Scott Paper Ltd. [4]. MeadWestvaco Corp. also has hybrid poplar plantations, of which the one in Maine was the source of the clones utilized in this study.

A major drawback of hybrid poplars is that the fiber and papermaking properties are different for each clone. Also, the fiber length for a specific clone (at the same age) can vary significantly with the site on which it grows [5]. This variation requires that the fiber and papermaking properties be analyzed for each plantation. With luck, it won't be long before some elite clones are identified with consistently high growth and survival rates and with chemical and physical properties well-suited for papermaking. Research on the identification of elite biomass producers is yielding encouraging results [6,7]. Also, research on characterizing the papermaking properties of hybrid poplars has been increasing in recent years [4,5,8,9].

Our objective was to compare the papermaking properties of kraft and mechanical pulps from four hybrid poplars to those of aspen (*Populus tremuloides*) and balsam poplar (*Populus balsamifera*). All the poplars were harvested within close proximity.

MATERIAL AND METHODS

Wood supply

All the poplars were harvested in Maine. The four hybrid poplars were (a) a 6 year-old DN 74 (*P. deltoides* × *P. nigra*), (b) a 7 year-old NE 19 (*P. nigra var. charkowiensis* × *P. nigra var. caudina*), (c) a 7 year-old NM 6 (*P. nigra* × *P. maximowiczii*), and (d) a 7 year-old OP 367 (*P. deltoides* × *P. nigra*). The aspen and balsam poplar were both more than 25 years old.

Kraft pulping

Kraft pulping was conducted in pressurized digesters (M&K) with liquor circulation. A 5:1 liquor-to-wood ratio was used with 16% active alkali (as Na_2O) and 28% sulfidity. The time to reach a maximum temperature of 165°C was 90 min, and the duration at that temperature was 120 min.

Kraft pulp bleaching

In the D_0 stage, the pulp was bleached in plastic bottles at 3.0–3.5% consistency at 70°C for 2 h, with an initial pH of about 3.5 (before the addition of ClO_2) and a final pH of about 2.3. A kappa factor of 0.20 (% equiv. Cl_2/kappa no.) was used on all occasions.

In the EP stage, we bleached the pulp in plastic bags at 12% consistency at 80°C for 2 h. The bleach charge was 2.0% NaOH, 0.25% H_2O_2, and 0.1% $MgSO_4•7H_2O$ on pulp. The final pH was always in the range of 11.2–11.6.

In the D_1 stage, the pulp was bleached in plastic bags at 10% consistency at 70°C for 3 h. The bleach charge was 0.8% ClO_2 and 0.3% NaOH on pulp. The final pH was always in the range of 3.5–4.1.

Mechanical pulping

The chips were treated with atmospheric steam for 8 min, then with 120°C saturated steam for an additional 3 min. The pressure was released, and primary-stage refining was conducted in a 12 in. KRK refiner with no applied steam pressure and with atmospheric discharge. The discharge consistency was about 25%. Refining in secondary and tertiary stages was conducted at about 15% consistency with atmospheric discharge.

Mechanical pulp bleaching

The pulps were pretreated with 0.2% sodium diethylene triamine pentaacetate (Na_5DTPA) and 2.0% SO_2 (from $NaHSO_3$) for 1 h at 90°C and 3% consistency. The pulps were then bleached with 3.0% H_2O_2, 3.0% NaOH, 2.0% sodium silicate, and 0.5% $MgSO_4•7H_2O$ at 60°C and 15% consistency for 4 h.

Analyses

TAPPI methods were used to determine wood density (T 258), ethanol and benzene extractives (T 204), total lignin (T 222), kappa number (T 236), viscosity (T 230), and strength (T 220). Optical properties were determined on an Elrepho brightness meter. A Kajaani FS 100 analyzer was used for coarseness and mass-weighted average fiber length found according to Eq. 1:

	Age, years	Density, g/cm³	Extract-tives, %	Lignin, %
Aspen	>20	0.379	2.7	18.4
Bal. poplar	>20	0.337	4.0	18.9
DN 74	6	0.348	2.1	20.0
NE 19	7	0.339	2.6	23.4
NM 6	7	0.361	3.3	20.3
OP 367	7	0.306	1.5	22.8

I. Age, wood density, lignin, and extractives content of the poplars.

	Kappa no.	Screened yield, %	Rejects, %
Aspen	17.4	56.2	0.3
Bal. poplar	19.8	56.3	0.2
DN 74	18.5	52.6	0.5
NE 19	16.8	52.8	0.2
NM 6	17.5	52.5	0.5
OP 367	17.2	52.5	0.2

II. Kappa numbers and pulp yields.

	Free-ness, mL CSF	Sheet density, g/cm³	Fiber length,[a] mm	Coarse-ness,[b] mg/m	Tensile index, N•m/g	Strain, %	TEA, J/m²	Tear index, mN•m²/g
Aspen	503	0.834	0.92	0.115	109	3.4	156	7.6
Bal. pop.	504	0.822	0.94	0.108	94	3.3	130	8.8
DN 74	395	0.923	0.77	0.112	105	4.4	204	7.4
NE 19	406	0.900	0.65	0.110	100	4.6	206	7.7
NM6	474	0.886	0.78	0.111	105	4.0	186	8.3
OP 367	434	0.860	0.65	0.113	102	4.4	201	7.7

[a]According to Eqation 1. [b]Unrefined fibers.

III. Physical properties of refined poplar kraft pulps.

	Final brightness, %	Final viscosity, cP
Aspen	90.7	24.1
Bal. poplar	90.5	22.6
DN 74	91.4	22.2
NE 19	91.6	23.9
NM 6	91.4	27.3
OP 367	90.4	23.7

IV. Bleachability of kraft pulps by $D_0(EP)D_1$ sequence.

$$L_w = \frac{\sum n_i L_i^3}{\sum n_i L_i^2} \quad (1)$$

where
i = 1, 2, 3, ...
n = fiber count
L = contour length.

RESULTS AND DISCUSSION

Wood properties

Table I presents data for three important wood properties—basic density, lignin, and extractives. Basic wood density is found by dividing the oven-dried weight by the water-swollen volume. It is important because the mass of chips treated in a digester or pretreatment vessel of fixed volume increases with wood density. The mass flow rate of chips should be as high as possible because pulp is sold on a weight basis. The disks that were used for determining density were taken at breast height (1.3 m above ground).

Low extractives and lignin contents are normally viewed as being favorable in commercial pulping and bleaching operations. As the table shows, each of the hybrid poplars had a lower basic density than aspen has, and each contained more lignin.

Kraft pulping results

As explained, all six poplars were cooked under similar conditions. As **Table II** shows, there was not a significant variation in kappa number (17.2 to 19.8), but the four young hybrids had significantly lower pulp yields than obtained with aspen or balsam poplar. Apparently, most of the hybrid poplars grown in the northeastern [10] or north central [11] regions of North America tend to have a lower cellulose or glucan content than aspen has and tend to produce kraft pulp at a lower yield.

Twenty pure and hybrid poplars, each grown on three sites in the north central U.S. [7], and two other poplars that survived on only two sites (64 samples) are presently being evaluated in our laboratory [12]. Most of those poplars (7.5 years) also have lower cellulose and higher lignin content than the mature aspen has. Interestingly, a higher percentage of hybrid poplar clones grown in the northwestern U.S. and western Canada have a cellulose content of about 49% [5,8], which is the approximate value for aspen [8].

In our lab, an excellent correlation between cellulose content and kraft pulp yield was observed [12], which is consistent with previous research [5,13].

Kraft fiber and sheet properties

Sheets were made after the six pulps were refined 1000 revolutions in a PFI mill (light load). Selected properties are presented in **Table III**. As the table shows, all four hybrid poplars in this study resulted in high sheet density, high percent strain (to the point of sheet rupture), and high tensile energy absorption (TEA), as previously reported for the DN 30 (*P. deltroides* × *P. nigra*) hybrid poplar [9].

The TEA is derived from the area under the stress–strain curve. The microfibril angle (MFA) is the average angle of the cellulose microfibrillar helix in the S2 layer of the cell wall, relative to the fiber axis [14]. When a tensile load is applied to sheets from refined hardwood kraft pulps, the percent strain increases with MFA [9,12,15,16]. This increased strain occurs at a high stress level and results in a significantly higher TEA.

Gurnagul *et al.* measured the MFA for kraft pulps from six different hardwood species [15]. The percent strain was interpolated at a tensile index of 95 N•m/g for all six pulps, both unbleached and bleached. Correlations were presented showing increasing strain with an increase in microfibril angle for both unbleached and bleached pulps. Although not reported, the R^2 value was at least 0.90 on both occasions.

The MFA has been reported to decrease with age for at least six different hardwoods—eucalyptus [14], eastern cottonwood [17], oak and beech [18], birch [19], and maple [20]. The data for eastern cottonwood [17] are significant to our study since clones of eastern cottonwood (*Populus deltoides*) are involved. The MFA of an eastern cottonwood was reported to decrease from 22.3° at the first growth ring to 12.7° at the twenty-fifth growth ring [17].

In all probability, if the MFA is averaged over seven years (hybrid poplars), a higher value will be obtained than if it is averaged over 25 years or greater (aspen and balsam poplar).

The higher sheet density of the DN 30 kraft pulp (compared to aspen kraft) was attributed in a previous study to a lower cell wall thickness [9]. All four hybrid poplars analyzed in the previous study were found to have thinner cell walls than aspen has. From the significantly higher sheet densities found in the current study, it appears that the fibers of all four of these hybrid poplars also have thinner cell walls than found in the fibers of aspen and balsam poplar.

The balsam poplar with the highest average fiber length afforded the highest tear strength. The overall results for NE 19 and OP 367 are particularly impressive when their low average fiber length (0.65 mm) is taken into account. The average fiber length for each unrefined pulp was higher than that of the refined sample by 0.0–0.1 mm. There were no significant differences in fiber coarseness.

Bleachability of kraft pulps

After a standard $D_0(EP)D_1$ treatment, all of the poplars attained an Elrepho brightness higher than 90%, as shown in **Table IV**. Pulp viscosity was greater than 18 cP for all the bleached pulps, which normally indicates a high degree of polymerization for the cellulose.

Two of the clones (NE 19 and NM 6) may be competitive with aspen or superior to it in terms of the overall properties expected for a bleached hardwood kraft pulp. The fiber lengths of these clones should increase by about 0.1 mm with two years of additional growth [8]. The advantages the clones will have in strength properties should be even more significant. The NM 6 clone, with a wood density approximately equal to that of aspen (Table I), had a higher final brightness and a higher viscosity than aspen had.

The NE 19 gave a lower kappa number after pulping and was bleached to a higher final brightness. If its lignin is indeed more reactive than that of aspen in pulping and bleaching, then it may be possible to decrease retention time in the digester, particularly during the impregnation stage. The lower basic density of NE 19 indicates that it is more porous than aspen. A lower retention time would increase the mass flow rate through the digester to compensate for the lower wood density.

	SRE, kW•h/ton	Freeness, mL	FIBER CLASSIFICATION				
			R28[a]	R48	R100	R200	T200[b]
Aspen - 2Sc	2972	88	—	—	—	—	—
Aspen - 3S	3732	33	16.3	22.2	20.0	16.6	24.9
Bal. poplar	4042	61	13.1	28.8	22.9	9.0	26.2
DN 74	4023	71	6.7	30.8	19.6	10.7	32.2
NE 19	3905	91	0.2	16.5	34.6	10.6	38.1
NM 6	3943	68	4.9	27.4	19.9	11.7	36.1
OP 367	3850	59	0.9	19.0	29.0	12.5	38.6

[a] Retained on a 28-mesh screen. bThrough a 200-mesh screen (fines).
[c] Two stages of refining; three stages for all other pulps.

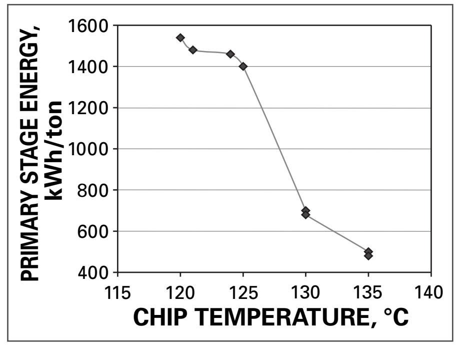
V. Characterization of unbleached mechanical pulps after two or three stages of refining.

Mechanical pulping

Primary-stage refining was performed by preheating the chips with atmospheric steam for 8 min, followed by pressurized steaming at 200 kPa (about 120°C) for 3 min. The pressure was released, and refining was conducted with atmospheric discharge. The process could be described as refiner mechanical pulping (RMP) because atmospheric discharge was used, but it could also be described as thermomechanical pulping (TMP) because the chips were preheated above 100°C before refining. **Table V** gives the specific refining energy for two or three stages of refining, the freeness, and the Bauer–McNett fiber classifications for the unbleached pulps.

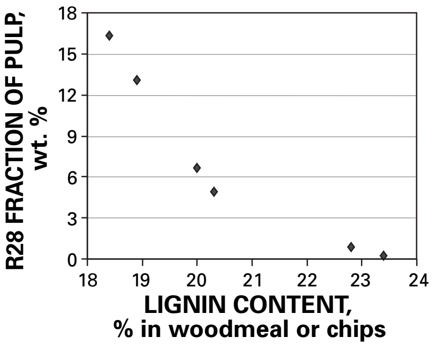
The aspen and balsam poplar had more long fibers (R28) and less fines (T 200) than the hybrid poplars exhibited. The low R28 values indicate that all the chips were very rigid when they entered the refining zone. Lignin is primarily responsible for chip stiffness under the refining conditions because the operating temperatures exceed the glass transition temperature of the cellulose and hemicelluloses [21–23].

Lignin is the amorphous polymer that binds wood fibers together, and the glass transition temperature (GTT), or the softening temperature of an amorphous polymer, is the temperature at which it becomes a deformable plastic. Wood is a viscoelastic, natural polymeric material whose stiffness (or torsional modulus) is greatly affected by temperature, moisture, and the rate of the loading of mechanical strain—*i.e.*, the frequency [21,22]. The frequency of bar contact with fibers and fiber bundles during the manufacture of RMP and TMP is extremely important [21,22]. Bars are the elevated surfaces on a metallic refiner plate. There is an upward shift of about 10°C in the GTT of wood for each order of magnitude increase in the frequency of bar contact [21]. The GTT of Norway spruce (*Picea abies*) increases from 90°C at 1.0 Hz [23] to 120–135°C [24,25] in a refiner where the frequency of bar contact is in the neighborhood of 1×10^4 Hz [21].



1. Estimation of the glass transition temperature of Norway spruce lignin under chip refining conditions.

We estimated the GTT of Norway spruce lignin under our refining conditions by preheating the chips for a long enough time for the temperature of the entire chip to approach the steam temperature. According to the solved differential equation for unsteady state conduction of heat in a wood chip [26], the temperature of the entire chip should be no more than 1C° or 2C° below the steam temperature after 30 min. Therefore, we preheated the chips for 30 min at predetermined temperatures and then refined them. All other variables, including the refiner plate clearance, were kept constant. As **Fig. 1** shows, increasing the temperature from 120°C to 125°C resulted in only a small



2. Long fiber fraction (R28) of mechanical pulp vs. lignin content (unextracted basis) of the poplar chips from which the pulp was produced.

	Unbleached brightness[a], %	H_2O_2 consumed[b], %	Bleached brightness, %	LAC[c], m²/kg	LSC[d], m²/kg
Aspen-2S	70.3	1.44	82.1	1.42	72.6
Aspen-3S	69.0	1.63	83.2	1.28	75.5
Bal. poplar	58.9	1.64	79.8	2.00	78.1
DN 74	69.6	1.47	83.7	1.18	74.6
NE 19	64.1	1.48	80.4	1.77	74.1
NM 6	68.8	1.61	82.5	1.41	76.1
OP 367	67.6	1.55	82.9	1.34	75.8

[a] After Na_5DTPA pretreatment. [b] Percent on pulp. [c] Light absorption coefficient. [d] Light scattering coefficient.

VI. Optical properties of unbleached and bleached RMPs/TMPs.

	Freeness, mL CSF	Sheet density, g/cm³	Tensile index, N•m/g	Strain, %	TEA, J/m²	Tear index, mN•m²/g
Aspen-2S	74	0.477	35.4	1.54	21.7	3.5
Aspen-3S	33	0.576	41.8	1.85	30.5	3.5
Bal. poplar	55	0.565	38.2	1.94	31.8	4.5
DN 74	53	0.485	32.8	1.84	26.2	3.5
NE 19	82	0.471	28.6	1.62	20.6	3.2
NM 6	62	0.568	32.7	1.61	22.0	3.4
OP 367	44	0.538	36.3	1.74	28.1	3.2

VII. Freeness and strength properties of bleached RMPs/TMPs.

decrease in chip rigidity, as indicated by the primary-stage refining energy. However, going from 125°C to 130°C resulted in a drastic loss in chip rigidity. This loss in rigidity can only be explained by the lignin's GTT being exceeded. The estimated GTT of 128°C falls with the expected range [24,25].

The GTT of aspen lignin under our refining conditions was estimated to be in the range of 115–120°C. A GTT that is lower for aspen than for Norway spruce is consistent with other reported data [22,23]. At a temperature about 5C° lower than the GTT, the lignin is still stiff, and fiber separation or fracturing occurs in an uncontrollable manner, leading to more broken fibers and a high fines content [22]. As the GTT is approached, the lignin begins to soften, and more fracturing occurs in the lignin-rich regions of the cell wall, such as the primary wall and middle lamella [22]. More long and intact fibers are obtained [22].

In our case, the chips were preheated with 120°C steam for 3 min, during which they probably attained an average temperature of 110–115°C. However, the system was at atmospheric pressure on both the feed and discharge sides during refining. The steam generated would likely cool the entering chips to an average temperature of about 110°C. At temperatures near 110°C, the lignins in all the poplars were still stiff and were acting as binder. More lignin in the hybrid poplars (Table I) meant stiffer chips, stiffer fiber bundles entering the refining zone, and more fiber breakage during refining. It seems unlikely that the variation in long fiber content (R28 fraction) could be explained only by the modest variation in lignin content (18.4% to 23.4%) of the chips. However, the correlation between the two variables, illustrated in **Fig. 2**, cannot be ignored.

Optical properties of mechanical pulps

Table VI presents the values for the unbleached and bleached brightnesses of the various pulps. Also included are data for the H_2O_2 consumed in bleaching and the light absorption coefficient and the light scattering coefficient for the bleached pulps (measured at 457 nm on an Elrepho brightness meter).

Instead of optimizing the alkali application for each pulp, we determined the brightening effect and H_2O_2 consumed for the same alkali consumption. If 3% H_2O_2 on chips were to be used in an alkaline peroxide mechanical pulping process for a poplar, then the alkali requirement would be about 5% NaOH on pulp. We applied 3% H_2O_2 and only 3% NaOH to the pulps (plus 0.23% NaOH from the 2% sodium silicate that was included in the formulation). The result was full alkali consumption (final pH of 7.1–7.5) for all samples.

Table VI shows that all the hybrid poplars except NE 19 had optical properties equal to those of aspen. The center of the NE 19 bolewood was significantly darker than that of the other hybrid poplars. The high values for the light scattering coefficient would indicate that our 12 in. refiner was quite competent for simulating RMP and TMP manufacture.

Physical properties of bleached mechanical pulps

The freeness and strength properties of the bleached pulps are presented in **Table VII**. The strength properties of all the hybrid poplar pulps were quite impressive when their fiber classifications were taken into account. Even though they contain much less of the long fibers than the three-stage aspen pulp contains (Table V), the tear indices for DN 74 and NM 6 approached that of aspen.

The NE 19 and OP 367 pulps had identical tear indices that were only 10% lower than for aspen pulp. These tear indices were high when you consider that these pulps contained only a very small amount of long fibers. The R28 fraction for the aspen pulp was 16.3%, whereas these two hybrid poplar pulps contained less than 1.0%.

The balsam poplar had high strength values, but this particular bolewood was very dark and is not suited for mechanical pulps. It afforded the lowest brightness, whether bleached or unbleached.

CONCLUSIONS

The four hybrid poplar kraft pulps had tensile properties superior to those of aspen and balsam poplar. The high densities for the sheets derived from the hybrid poplar pulps indicated that the cell walls of these fibers are not as thick as those of the two traditional poplars. The high strain and TEA of the sheets made from the hybrid pulps indicated that the fibers of these hybrids have a higher average microfibril angle. The hybrid poplars were much younger than the traditional poplars. These results are consistent with an earlier study of some other hybrid poplar clones [9].

All six poplars were pre-heated and refined similarly in the manufacture of RMP/TMP. However, the aspen and balsam poplar afforded pulps with significantly more long fibers and higher strength values. Three of the four hybrid poplar pulps had bleached brightnesses equivalent to that of aspen, while the other hybrid poplar and the balsam poplar had much lower brightnesses and were unsuitable for mechanical pulp grades. If refining were to be optimized so that the fiber classifications were similar, we would project that hybrid poplar mechanical pulps would be stronger than a corresponding aspen pulp. **TJ**

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